

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: Colin Robert Willis *et al.* ) Confirmation No. 5079  
Application No.: 10/018,727 )  
Date Filed: August 9, 2002 ) Art Unit: 1792  
For: FUNCTIONALISED SOLID SURFACES ) Examiner: PADGETT,  
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Commissioner of Patents

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Attorney Docket No. 41577/266144

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*Melody J. Wilson*  
Melody J. Wilson

**DECLARATION UNDER 37 C.F.R. § 1.132 BY STEPHEN COULSON**

I, Stephen Coulson, do hereby declare:

1. I hold a Ph.D. from the University of Durham in surface chemistry, and I have worked in the field of pulsed plasma deposition of thin polymer films for over 12 years. I have a number of publications and patents covering low average power deposition under pulsing conditions to maximize structural retention of starting chemicals. From September 1996 - October 1999, I was working in the laboratories of the University of Durham where the work reported in the present patent application was carried out. I was a member of the group that carried out this work which was a direct result of the research I carried out. I am the founder of P2i, a company specializing in plasma deposition of coatings onto a variety of substrates, and I have been P2i's R&D Director for five years. P2i is a licensee of the subject matter claimed in the present application. I have read the present patent application (U.S. Serial No. 10/018,727), the Office Action mailed on August 13, 2008, and the references discussed therein.

2. I conclude, based on the analysis presented below, that the claimed methods differ substantially from the methods described in Timmons *et al.* (U.S. Patent No. 5,876,753) ("Timmons") and that one of ordinary skill in the art would not have found the claimed method to be obvious in view of the teachings of the cited prior art, and in particular the Timmons reference.

3. The Examiner has identified that it is difficult to make true comparisons between the pulsed plasma polymerization conditions that characterize the claimed method and those conditions which are disclosed in Timmons. Whereas we have used as the defining parameter the average power **density**, Timmons' data is in the form of average powers. Moreover, because Timmons does not state the volume of the chamber in which the average power is applied, the plasma density cannot be calculated; therefore direct comparison of power densities is not possible. The Examiner has also pointed out that the experimental data in the present application refers only to peak powers for the generation of the plasma. However, the Examiner has herself used a formula that is provided in Timmons for deriving average powers on the basis of given peak power data. The use of this formula will allow proper comparisons to be made between Timmons and the claimed method.

4. By the use of the Timmons formula, the Examiner has been able to derive average power data from the Timmons patent and states on page 6 lines 3-5 of the Office Action of August 13, 2008 that "average powers for exemplary duty cycles [in Timmons] would be approximately 75W, 33W, 12.5W and 0.095W respectively, but are for total power output, not power density, thus cannot be directly compared to the claimed parameter range" [viz. an average power density of not more than 0.0025W/cm<sup>3</sup>]. It is noted that the final number provided by the Examiner is believed to be incorrect and should instead be **9.5W** (*i.e.*, 3/63 x 200W).

5. Although the chamber volume used is not listed in the present patent specification, I can confirm that it was 470cm<sup>3</sup>. Consequently, the claimed range for the average power density parameter of less than 0.0025W/cm<sup>3</sup> equates to a maximum

average power of  $470 \times 0.0025 = 1.175\text{W}$ . Or, on a like-for-like basis, the maximum possible power used in the process described in the present application is **almost an order of magnitude less than** the lowest value for this parameter that can be derived from Timmons.

6. The examples that are set out in the present patent application underline further the substantial differences between the respective processes. In the examples, a peak power of 40W is used, with a time “on” of  $20\mu\text{s}$  and a time “off” of 20ms. Using the formula given in Timmons, this means that the average power was just under 0.04W which is **more than 200 times less than any average power referred to in Timmons**.

7. Further experiments have now been conducted with glycidyl methacrylate (GMA) which demonstrate that it is **only at extremely low power densities, combined with a specific choice of a monomer containing a functional group that will polymerize easily**, that full retention of the epoxy functionality is achieved. This is important for maximizing the subsequent reactions for achieving the best possible performance of the resulting product. Two plasma polymerizations were carried out at respective average power densities of 0.0025 and  $0.00025\text{W/cm}^3$  with the following analytical data for the polymer product obtained by XPS:

Carbon Environment	Theoretical (%)	Power density $0.00025\text{W/cm}^3$ (%)	Power density $0.0025\text{W/cm}^3$ (%)
Epoxide	28.6	25.01	18.35

These results show that **just outside the claimed limit of average power density of  $0.0025\text{W/cm}^3$  there is observed a substantial falling off in the percentage of the epoxide functionality of GMA that is retained in the plasma polymer obtained**, especially when viewed in the light that a practically realizable maximum epoxide content for polymerized GMA is in fact most likely to be somewhat lower than the theoretical figure. By dropping below the claimed limit of power density in

accordance with the claims of the present application, it is clear that far better structural retention results, which is the desirable outcome.

8. The Examiner asserts that the establishment of our reaction conditions was merely a matter of "routine experimentation to provide an effectively low power plasma as taught by Timmons *et al.* to enable retention of active functional groups." However, while Timmons certainly refers to the use of "low" powers, the patent does not define how such "low powers" are to be judged, and all of the evidence in the Timmons patent specification points towards average powers (in W) for the polymerization process which are of the order of no lower than single figures (e.g., 9.5W as discussed above). In my opinion, the reaction conditions of the present application are completely remote from anything that was contemplated by Timmons *et al.* and well outside of the scope of "reasonable experimentation" based on the evidence in the Timmons patent. It also requires a judicious choice of starting chemical with a particular polyermizable group.

9. In my view the Office Action does not explain how Timmons can be read as teaching the use of the presently claimed very low average powers and the specific choice of a monomer containing a functional group that will polymerize easily. In my opinion **is it not a simple matter of routine experimentation** to determine that such very low powers were necessary in order to achieve the desired functional retention as demonstrated by the applicants of the present application in producing a plasma polymer from GMA.

10. Here it is also appropriate to point out that Timmons did not address the use of GMA monomer as the feedstock for his process, consequently the teachings of Timmons are not necessarily a useful guide to how GMA can best be processed to obtain a polymer with high retention of the epoxy functional group. Indeed, some experiments were performed by the applicants of the present application (reported as Examples 4 and 5 in the present patent application) which are very suggestive of the fact that Timmons is not at all a useful guide on this point.

11. Examples 4 and 5 demonstrate that at least one epoxy-containing material (allyl glycidyl ether (AGE)) that is exemplified in Timmons did not, in fact, provide an effective coating when laid down under the conditions set forth in the present application. Not only was the rate of deposition found to be very slow but also a **substantial proportion of the epoxy functionality was lost in the resultant polymer layer.** Against this background, the discovery that GMA was very successfully deposited under **identical conditions** was, in my view, surprising and unexpected.

12. My point here is, I believe, underscored by the Examiner herself in the discussion at paragraph 8 of the Office Action dated August 13, 2008, to quote:

As noted above Timmons et al teach allyl glycidyl ether (AGE) as an **exemplary epoxy**-containing monomer, instead of the claimed epoxy compound of GMA, but **Kolluri et al** also teach analogous plasma polymerization reactions, where either **AGE or GMA** are taught to be **deposited for their reactive epoxy functional groups**, hence use of GMA in Timmons et al would have been expected to be effective for the taught process due to taught equivalent usage, providing an alternative species for the generically taught epoxy containing monomers.

In my opinion, the evidence of the comparative examples clearly shows that there is no such equivalent usage nor can there be said to be any class of “generically taught epoxy containing monomers,” which I find not at all surprising given that Kolluri (US Patent No. 5,723,219) again uses significantly higher powers than those of the present application.

13. I acknowledge that it could be argued that both the Kolluri reference discussed above and the Connell patent (GB1,037,144) suggest that both AGE and GMA form ‘equivalent’ members of any generic group of epoxy-containing monomers. However, I note that none of the cited references provide an illustrative worked example of the deposition of either monomer, nor indeed of any epoxy-containing monomer. The problems associated with the deposition of such compounds reported

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in Examples 4 and 5 of the present application could not have been appreciated. The fact that these problems do not apply to GMA appears to me therefore to represent a significant scientific contribution.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

13/2/09  
Date

Stephen Coulson  
Stephen Coulson